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U.S. ATOMIC ENERGY COMMISSION

REGULATORY GUIDE

DIRECTORATE OF REGULATORY STANDARDS

REGULATORY GUIDE 5.39

GENERAL METHODS FOR THE ANALYSIS OF URANYL NITRATE SOLUTIONS FOR ASSAY, ISOTOPIC DISTRIBUTION, AND IMPURITY DETERMINATIONS

A. INTRODUCTION

Paragraph 70.22(b) of 10 CFR Part 70, "Special Nuclear Material," requires, among other things, that an applicant for a license to possess certain quantities of special nuclear material in an unsealed form describe his procedures for the control of and accounting for special nuclear material. This guide identifies methods acceptable to the Regulatory staff for chemical, isotopic, and impurity analyses which an applicant may specify as part of his procedures for accounting for special nuclear material.

B. DISCUSSION

Committee C-26 on Fuel, Control, and Moderator Materials for Nuclear Reactor Applications of the American Society for Testing and Materials (ASTM) has developed a standard containing methods for the chemical analysis of nuclear-grade uranyl nitrate solutions. This standard is entitled "Standard Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Uranyl Nitrate Solutions."* As used in this standard, *nuclear grade* material means material that is to be used *exclusively* for the fabrication of nuclear fuel. Included in this standard are two methods for the determination of uranium:

1. Ferrous Sulfate Reduction - Potassium Dichromate Titration. The optimum quantities of uranium to be determined are 100 to 150 mg with a stated precision of 0.1 percent relative standard deviation (RSD) at the 2 sigma level.
2. Ignition (Gravimetric) Method. The stated precision is 0.03 percent RSD at the 2 sigma level for 5 to 10g of U_3O_8.

This standard also includes a method for the determination of the isotopic composition of $UO_2(NO_3)_2$ solutions allowing for the use of reference standards to determine bias. The stated precision of the method for ratios at the 0.1 isotopic concentration level is 0.02% RSD; at the 0.01 isotopic concentration level, 0.5% RSD; and at the 0.001 isotopic concentration level, 1.2% RSD, all at the 2 sigma level. Various impurities such as chromium, molybdenum, thorium, phosphorus, silicon, chloride, fluoride, bromide, iodide, nitrogen, carbon, sulfur, boron, and volatile and nonvolatile impurities can be determined using the methods that are included in the standard. Also described are methods for the determination of specific gravity, free acid, impurities by spark source mass spectrometry, U-232 by alpha spectrometry, total alpha activity, and fission product activity.

C. REGULATORY POSITION

The analytical methods for the measurement of nuclear-grade uranyl nitrate solutions contained in the ASTM Standard C-799, "Standard Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Uranyl Nitrate Solutions"* are acceptable and provide an adequate basis for the assay, isotopic measurement, and impurity analysis of nuclear-grade uranyl nitrate solutions subject to the following:

1. Precision and Accuracy Statements. The listed statements provide guidance to the levels of performance which may be attained using the described methods. The actual precision and accuracy of a method applied within a selected laboratory can only be determined through a well-planned measurement control program.
2. Calibration and Standardization. The standards should be prepared in the same matrix as the samples, and the calibration points should bracket the estimated range of the samples.

*Copies may be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pa. 19103.

3. Safety. Procedures involving the use of perchloric acid should be performed with caution because of the potential explosion and fire hazard.

D. IMPLEMENTATION

The purpose of this section is to provide information to applicants and licensees regarding the Regulatory staff's plans for utilizing this regulatory guide.

This guide reflects current regulatory practice. Therefore, except in those cases in which an applicant or licensee proposes an alternative method for complying with specified portions of the Commission's regulations, the method described herein will be used immediately in the evaluation of applications for licenses to possess at any one time special nuclear material in a quantity exceeding one effective kilogram as described in §70.22(b) of 10 CFR Part 70 and in the evaluation of the performance of holders of such licenses..